

Reactivity of Thin Metal Films on Sodium Beta" Alumina Ceramic in High Temperature, Low Pressure Sodium Vapor

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Abstract. Electrochemical techniques including impedance spectroscopy are routinely used to test the performance of AMTEC electrodes. These experiments may be carried out in an actual AMTEC cell, or in an sodium exposure test cell SETC where the sodium pressure and the temperature are identical at both the anode and cathode. These tests reveal details of electrode material grain growth, electrode decomposition and reaction of electrode materials with the sodium beta" alumina solid electrolyte. Tests of the same sort may be used to examine the compatibility of metals and sodium beta" alumina under simulated AMTEC operating conditions. Preliminary tests comparing rhodium/tungsten, molybdenum, titanium and vanadium electrodes are reported.

INTRODUCTION

The Alkali Metal Thermal to Electric Converter (AMTEC) is a direct energy conversion device capable of near-Carnot efficiencies; its general characteristics and major features are described in detail elsewhere (Cole, 1983; Hunt et al, 1975, 1978, 1981, 1982; Ryan et al, 1994a, 1995; Vining, et al, 1993; Underwood et al, 1992a, 1992b; Weber, 1974; Williams et al, 1990a, 1990b) NASA's interest in AMTEC is as the thermal-to-electric power conversion device for outer planet exploratory spacecraft, requiring an operating lifetime of 7 - 15 years. Our goal has been the prediction of life behavior of critical AMTEC components using accelerated testing through the use of higher temperatures and more extreme chemical environments. Our accelerated tests are verified by tests at more typical AMTEC operating conditions.

Applications of metallic films on sodium beta"-alumina solid electrolyte, BASE, in technology for the alkali metal thermal to electric converter, AMTEC, include both electrodes and structural components such as seals. (1) Both types of component must maintain their performance on BASE in a sodium atmosphere at about 1125K for the life of the AMTEC device. We have previously reported on the stability of the BASE electrolyte and several electrodes under AMTEC operating, or slightly accelerated, conditions. (Ryan et al, 1991, 1992, 1994b, 1994c, 1994b, 1998a, 1999a, 2000; Shieldset al, 1999; Williams et al, 1990c, 1991, 1993, 1995, 1998, 1999, 2000) While the requirements for metallic seals are very different than those for AMTEC electrodes, the interface between the BASE and the bonding layer of the metal seal may be investigated by using electrochemical techniques which are commonly used to characterize electrodes for AMTEC.

In addition, the stability of seal morphology and strength must also be investigated, and in general electrochemical methods are less useful than exposure tests for these purposes. Thin films on BASE are studied as is, or after brazing a metal foil to the metal film. Exposure chambers are evacuated stainless steel tubes with refractory metal liners such that the liner extends into the cooler part of the tube. A small amount of sodium metal is introduced into the tube before it is evacuated and sealed, and with the cool end of the tube controlled between 573 and 773K, provides a known pressure of sodium gas over the sample.

Evidence collected during previous studies indicates the chemical stability of Mo, W, WPt, and WRh films on BASE in sodium vapor for periods of up to 6800 hours, at temperatures as high as 1173K. Ti alloys resulted in

some degradation of the BASE surface, with inclusion of Ti, and both Cr and Mn led to decomposition of BASE with formation of Cr_2O_3 and MnAl_2O_4 as well as Al_2O_3 (Williams, 1998a, 1999).

Specific requirements for metals contacting BASE, and their effect on fundamental cell impedance characteristics are discussed below.

Metallization on BASE

Applications of metallic films on sodium beta"-alumina solid electrolyte (BASE) ceramic in technology for the alkali metal thermal to electric converter (AMTEC) include both electrode and metallization functions. Electrochemical characterization over the course of accelerated tests at up to 1173K for 6000 hours have demonstrated the durability of the best AMTEC electrodes, such as tungsten-rhodium alloys (Williams et al, 1989). The metallization of BASE for other purposes usually involves ultimate formation of a seal between BASE and a structural metal or ceramic component. Characterization of metallizations using the same electrochemical techniques should provide information about any degradation processes going on at the BASE/metal interface.

The Exchange Current, or Apparent Charge Transfer Resistance

B is a semi-empirical constant equal to the exchange current of an AMTEC electrode corrected to unit activity sodium gas, and normalized for the collision rate of sodium atoms at the surface. Experimentally, this constant fits the exchange current well for temperatures over 700-1200K. A fundamental model for the exchange current at Mo electrodes shows that B should give a good, but not perfect, fit to the exchange current from 600-1350K (Williams et al, 1990a, 1998b).

B is usually measured at an AMTEC cathode in low pressure to moderate pressure sodium gas, in an AMTEC cell with an anode in high pressure sodium gas or liquid sodium. The anode at high activity sodium has negligible impedance. Impedance spectra taken at a series of d.c. biases in AMTEC cells are best used to determine the exchange current, however AMTEC current-voltage curves may be used if other electrode parameters are known. It may also be determined from impedance spectra plus current-voltage curves taken in sodium exposure test cells, where both electrodes are at the same pressure. The exchange current has an approximate reciprocal relationship with the apparent charge transfer resistance of the electrode, R_{act} . The term apparent is used because the resistive element due to the true charge transfer resistance always contains some component, often small, due to resistance of sodium transport through the electrode. In many cases the cell may be regarded as being equivalent to R_{act} in parallel with the electrolytic cell capacitance, C_{ec} , with both of these quantities in series with an ohmic series resistance, R_{ser} .

The Electrolytic Cell Capacitance

The electrolytic cell capacitance, C_{ec} , will be proportional to the area of the BASE/electrode interface, and inversely proportional to the thickness of any insulating gap between the solid electrolyte and the electrode. Hence formation of an interfacial degradation layer which is an insulator will be easily observed in the electrolytic cell's capacitance, most easily determined from electrochemical impedance spectroscopy. Formation of electrochemically active degradation layers may give rise to a very large pseudocapacitance characterized by a very large time constant. (Williams et al, 1990b)

The Electrolytic Cell Series Resistance

The electrolytic cell series resistance, R_{ser} , will be modified by any reaction or morphology change which changes the electrical conductivity of the electrode or the ionic conductivity of the solid electrolyte. In particular, formation of an insulating layer at the BASE /electrolyte interface, such as occurs when $\text{Al}_2\text{O}_3/\text{Cr}_2\text{O}_3$ forms due to the reaction of BASE with chromium will lead to an extreme increase in the R_{ser} . When the reaction of BASE with a reactive metal leads to an insulating product more distributed throughout the BASE volume, as in the case of its reaction with Mn, the cell R_{ser} series resistance will rise, but not so extremely. Reaction of BASE with Ti or V, which may lead to electrically conducting and ionically conducting products, may be expected to be observable as some systematic change in the R_{ser} and the C_{ec} .

EXPERIMENTAL

Tests in low pressure sodium atmosphere are carried out in Sodium Exposure Test Cells, SETC's (Ryan, et al, 1988b), with refractory metal liners. These experiments can routinely be operated for duration of hundreds to thousands of hours with *in situ* monitoring of the electrical and electrochemical responses of the electrode and solid electrolyte. Current voltage curve measurements and Electrochemical Impedance Spectroscopy, EIS, were carried out routinely in all of these SETC tests. Other tests of chemical stability at high temperature in vacuum or low pressure sodium vapor, without electrical leads were carried out in high purity alpha alumina chambers, or in stainless steel chambers with refractory metal liners.

RESULTS AND DISCUSSION

The primary tests carried out to extend our understanding of electrodes on BASE in sodium atmosphere at high temperature was an experiment which compared two standard electrodes, molybdenum and rhodium/tungsten, with two refractory elements, titanium and vanadium, which might be used to promote adhesion of a braze seal material onto a BASE surface. All were deposited as thin electrodes, circa one micron thick, by magnetron sputtering, and were contacted by tied on molybdenum wire leads. The experiment was run as a typical SETC experiment, in a furnace which provided a nearly constant temperature hot zone extending over the four sample BASE tubes each with a different electrode material. After heat up to 1123K, this test was terminated prematurely due to a leak. Times will be given with respect to the time at which 1123K was first reached: the experiment had been at significantly elevated, but somewhat lower, temperatures for about 48 hours before this.

Initial electrode performance, which may be divided into series resistance and apparent charge transfer resistance, R_{act} , declined from rhodium tungsten, to vanadium, and to molybdenum, and finally most dramatically to titanium which gave very poor performance. The current voltage curves of all electrodes showed the initial presence of an electrochemically active (with respect to sodium ions) metal oxide, which decreased in the first few hours for all the electrodes except titanium. The vanadium electrodes lose most of their oxide component quickly at 1123K in low pressure sodium gas, and show good electrode behavior, suggesting adhesion and lack of a reaction layer, for initial eight hours. Surprisingly, vanadium was initially almost as good an electrode as rhodium tungsten, but its performance declined faster after oxide loss, almost certainly due to rapid grain growth. Figures 1 and 2 show the current voltage curves of rhodium/tungsten and vanadium electrodes after loss of much of the volatile oxides in the first hours at high temperature.

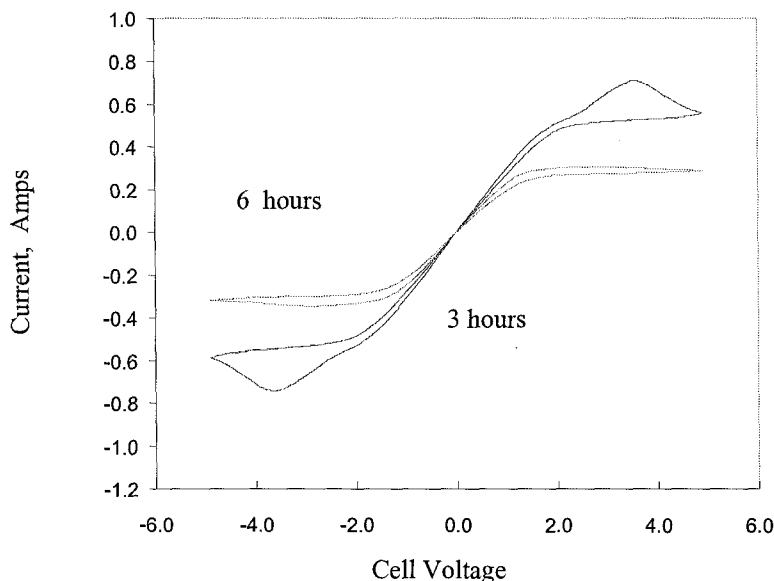


Figure 1. Current Voltage curves of rhodium tungsten electrodes at 3 and 6 hours at 1173K

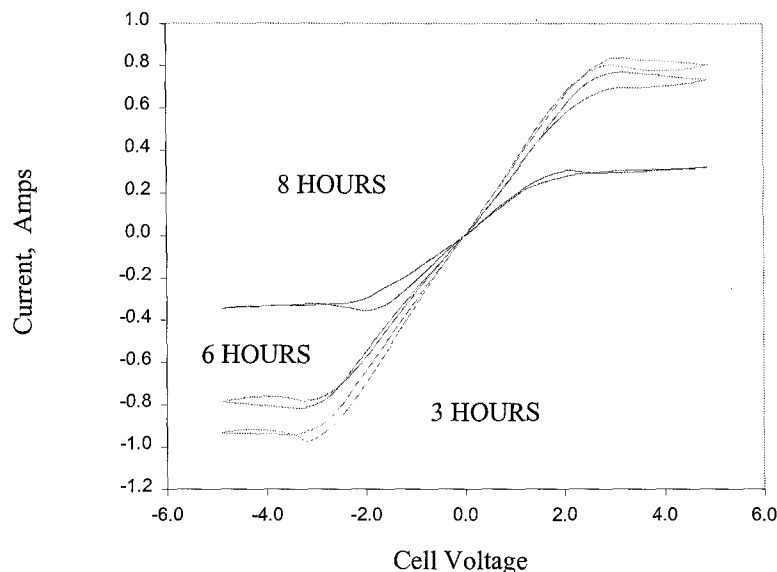


Figure 2. Current Voltage curves of vanadium electrodes at 3 and 6 and 8 hours at 1173K

The EIS responses showed low series resistances increasing from vanadium to rhodium tungsten to molybdenum, but all between 2 and 3 ohms. The titanium series resistance was about 16 ohms. The R_{act} of the titanium electrode was about five times higher than the next electrode, molybdenum. Vanadium's R_{act} was a factor of four times smaller than molybdenum's R_{act} and rhodium/tungsten's R_{act} was about 20 times smaller. The molybdenum electrode was somewhat poorer in performance than typical molybdenum electrodes, and the rhodium/tungsten electrode was comparable with our best rhodium/tungsten electrodes, which, however, show little variation. Both the high series resistance and the low R_{act} of the titanium electrode are consistent with poor electrical contact at the interface; however, the titanium electrode was quite adherent. We propose that an interfacial reaction layer between titanium and BASE formed, and acts as a poor electronic and ionic conductor. Titanium based brazes on BASE always show, and titanium nitride electrodes on BASE sometimes show an alteration zone (circa micron thickness) of gray to black material at the electrode/electrolyte interface. This material contains Ti, Al, O, and Na by XPS, but is poorer in Na than the solid electrolyte. Sodium ion transport through this layer may add an activated step or reduce effective charge transfer reaction area. This material also appears to be either an insulator or a poor electronic conductor.

All of the electrodes examined in this study have exhibited the presence of electrochemically active oxide phases. We interpret their behavior generally on the basis of extensive studies of molybdenum electrodes with and without added oxide phases (Wheeler, et al 1988, Williams et al 1986, 1988, 1990b). Molybdenum, tungsten and vanadium are all known to form volatile compounds when high metal oxidation state oxide reacts with sodium. In contrast, volatilities of sodium-titanium-oxygen compounds are all much less than those of, for example sodium molybdate or sodium meta- or ortho-vanadates. The presence of liquid molybdates, tungstates or vanadates also gives rise to a transient very high exchange current, most thoroughly examined for sodium molybdate/molybdenum electrodes (Williams et al 1986, 1988, 1990b). While the accuracy of the high exchange currents, (on the order of $B = 400-600$) measured for $Na_2MoO_4/Na_2Mo_3O_6/Mo$ electrodes is not high, it is clear that they are much better charge transfer electrodes than "clean molybdenum electrodes with exchange currents of about $B=120$. In TABLE 1 the exchange currents for all of the electrodes measured at JPL are presented for comparison. We propose that charge transfer occurs at the exterior surface of the electrode over a much larger reaction zone; specifically on Mo grains coated with Na_2MoO_4 and at the surface of $Na_2Mo_3O_6$.

The presence of titanium oxide in the titanium electrode is not likely to be detrimental; it should still enhance its transport properties and exchange current. (Ryan et al, 1999b) However, titanium metal is expected to react with BASE to form more complex non-volatile oxide phases. Ti braze alloys result in some degradation of the BASE surface, with inclusion of Ti, and similarly both Cr and Mn lead to decomposition of BASE with formation of non conducting oxides, Cr_2O_3 and $MnAl_2O_4$ as well as Al_2O_3 . We conclude, preliminarily, that vanadium may have

significant advantages over titanium as an adherence layer or promoter for seals of metals to BASE, especially in preventing long term interfacial degradation.

TABLE : Exchange currents observed for various electrodes and metal coatings. Only JPL results are included.

Electrode	Average B	Range of B(T)	Data set size/quality/method
Molybdenum	120	50-175(700-1225K)	Large/Excellent/ AMTEC - EIS and iV
Tungsten	High	50-100(1075-1225K)	Moderate/ Excellent/ AMTEC -EIS and iV
Platinum/Tungsten	80	50-100(900-1200K)	Moderate/ Good/ AMTEC - EIS and iV Large/ Good/ SETC- EIS and iV
Rhodium/Tungsten	120	90-150(1100-1200K)	Small/ Good/ AMTEC - EIS and iV Large/ Good/ SETC- EIS and iV
Titanium Nitride	60	30-90(1050-1123K)	Small/Good/ AMTEC - EIS and iV Moderate/Good/ SETC - EIS and iV
Mo/Na ₂ MoO ₄	500	400-600(1100-1200K)	Small/ Fair/ AMTEC - EIS and iV
W/Na ₂ WO ₄	High	200-300(1100-1225K)	Small/ Fair/ AMTEC - EIS and iV
Mo/TiO ₂	High	100-300(900-1250K)	Moderate/ Good/ SETC - EIS and iV
Titanium	Low	<20 (1173K)	Small/ Good /SETC - EIS and iV
Vanadium	High	about 100 (1173K)	Small/Good / SETC - EIS and iV

CONCLUSIONS

The same techniques which have been used to establish the stability characteristics of AMTEC electrodes on BASE at high temperature in low pressure sodium vapor can be used to perform a partial evaluation of metal braze and structural components which contact BASE. It is fairly well established that a number of useful electrode materials are quite adherent to BASE under operating conditions. This study shows that vanadium is also an adherent metal, with no tendency over the short times of about 10 hours to react with BASE even on a several Angstrom scale. Titanium metallization needs further testing but should be regarded a potentially damaging based on the current evidence.

ACKNOWLEDGMENTS

The research reported in this paper was carried out by the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration. Research was supported by NASA Code S.

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